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=> d l7
L7 HAS NO ANSWERS
L7                STR
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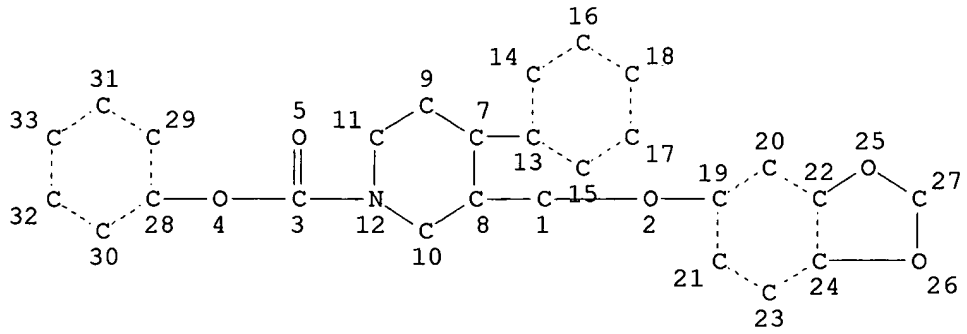

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DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS  32

STEREO ATTRIBUTES: NONE


=> s l7
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SAMPLE SCREEN SEARCH COMPLETED -        0 TO ITERATE

100.0% PROCESSED         0 ITERATIONS                                 0 ANSWERS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS:  ONLINE  **COMPLETE**
                        BATCH   **COMPLETE**
PROJECTED ITERATIONS:            0 TO         0
PROJECTED ANSWERS:               0 TO         0

L8             0 SEA SSS SAM L7

=> s l7 ful
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FULL SCREEN SEARCH COMPLETED -       19 TO ITERATE

100.0% PROCESSED        19 ITERATIONS                                6 ANSWERS
SEARCH TIME: 00.00.01

L9             6 SEA SSS FUL L7

=> fil caplus
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                       ENTRY    SESSION
FULL ESTIMATED COST                                   184.00     197.47

FILE 'CAPLUS' ENTERED AT 11:02:02 ON 07 MAR 2006
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FILE COVERS 1907 - 7 Mar 2006 VOL 144 ISS 11
FILE LAST UPDATED: 6 Mar 2006 (20060306/ED)

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=> s l9
L10         20 L9

=> s l10 not l1
L11         19 L10 NOT L1

=> d bib abs hitstr 1-19

L11  ANSWER 1 OF 19  CAPLUS  COPYRIGHT 2006 ACS on STN
AN   2002:736935  CAPLUS
DN   137:247681
TI   Novel process for the preparation of a carbamate, a key intermediate in
     the synthesis of paroxetine
IN   Lucas, Edward
PA   Smith Kline Beecham PLC, UK
SO   U.S. Pat. Appl. Publ., 7 pp., Cont. of U.S. Ser. No. 635,545, abandoned.
     CODEN: USXXCO
DT   Patent
LA   English
FAN.CNT 1
     PATENT NO.          KIND   DATE        APPLICATION NO.         DATE
     ---------------     ----   --------    --------------------    --------
PI   US 2002137938        A1    20020926    US 2002-109119          20020327
PRAI GB 1998-17195        A     19980807
     US 1999-370041       B1    19990806
     US 2000-635545       B1    20000810
OS   CASREACT 137:247681; MARPAT 137:247681
GI
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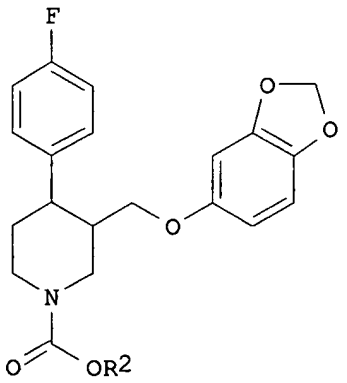

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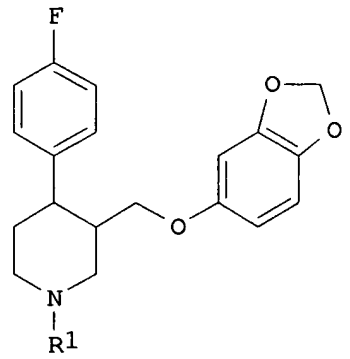


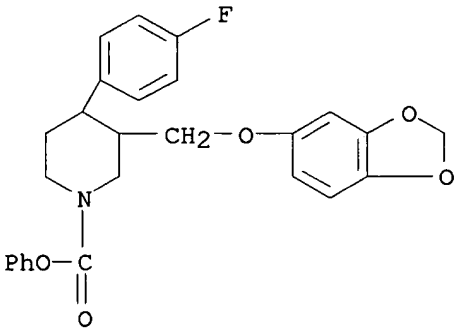
II

AB The title carbamates [I; R2 = alkyl, haloalkyl, cycloalkyl, aralkyl, (un)substituted aryl] were prepared by reacting a solution of a compound II [R1 = alkyl, arylalkyl, alkynyl] at 50-100°C with a haloformate HalCO2R2. Thus, reacting 4-(4-fluorophenyl)-1-methyl-3-(3',4'-methylenedioxyphenoxymethyl)piperidine with Ph chloroformate at 60-65°C afforded 88% I [R2 = Ph]. Such prepared compds. I can be then converted to paroxetine and its salts which is used in treating depression, obsessive compulsive disorder and panic.

IT **262424-80-6P**
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(novel process for the preparation of a carbamate, a key intermediate in the synthesis of paroxetine)

RN 262424-80-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester (9CI) (CA INDEX NAME)



L11 ANSWER 2 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 2002:314911 CAPLUS

DN 136:325429

TI Process of the preparation of 3-substituted-4-arylpiperidines useful as intermediates in paroxetine synthesis

IN Ward, Neal

PA Smithkline Beecham P.L.C., UK

SO PCT Int. Appl., 20 pp.
CODEN: PIXXD2

DT Patent

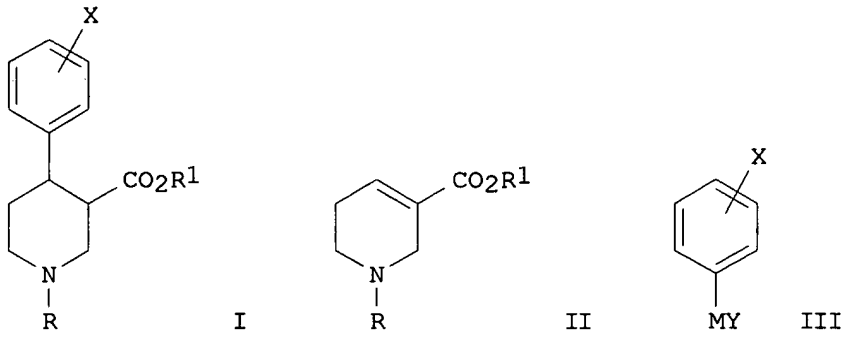
LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2002032870	A1	20020425	WO 2000-GB4071	20001020
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	AU 2001010390	A5	20020429	AU 2001-10390	20001020
PRAI	WO 2000-GB4071	A	20001020		

OS MARPAT 136:325429

GI



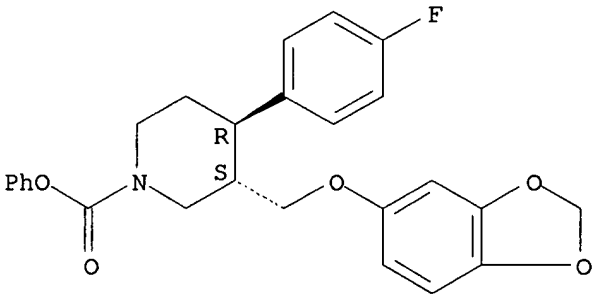
AB The title compds. I [R, R1 = alkyl, aryl, arylalkyl; X = H, halo, OH, alkoxy, etc.] which are important intermediates in the preparation of inter alia paroxetine, are prepared by reaction of an arecoline analog II with an organometallic compound containing an X-substituted Ph group, such as a compound III. Suitably the compound III is a Grignard reagent, where M is magnesium and Y is a halogen atom, or M may be a Group II metal and Y is a halogen atom or a second X-substituted Ph group. When III is a Grignard reagent, the reaction is carried out either in a suitable non-ether solvent, typically a hydrocarbon or a non-reactive chlorinated hydrocarbon, or in a mixture of such a solvent with di-Et ether. E.g., preparation of (±)-trans-1-methyl-3-carbomethoxy-4-(4'-fluorophenyl)piperidine (IV) by reacting 4-fluorophenylmagnesium bromide and arecoline followed by epimerization of the resulting cis/trans mixture, is described. A multi-step synthesis of paroxetine using IV is also presented.

IT **253768-88-6P**
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(process of the preparation of 3-substituted-4-arylpiperidines useful as intermediates in paroxetine synthesis)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

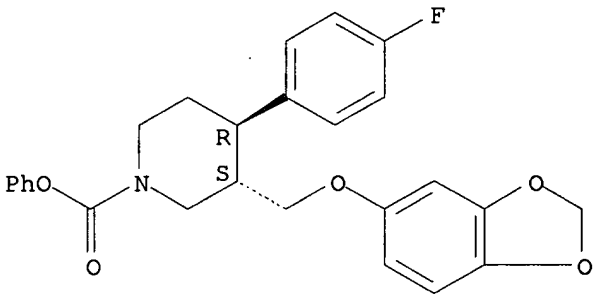
L11 ANSWER 3 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2002:138850 CAPLUS
DN 136:183712

TI Preparation and formulation of paroxetine methanesulfonate
PA Smithkline Beecham P.L.C., UK
SO Ger. Gebrauchsmusterschrift, 41 pp.
CODEN: GGXXFR
DT Patent
LA German
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI DE 20022646	U1	20020221	DE 2000-20022646	20001228
PRAI DE 2000-20022646		20001228		

AB The title compound was prepared and formulations comprising it were given.
IT **253768-88-6**, N-Phenoxycarbonylparoxetine
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation and formulation of paroxetine methanesulfonate)
RN 253768-88-6 CAPLUS
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



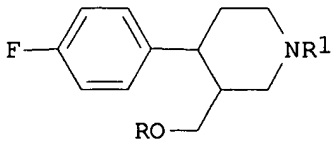
L11 ANSWER 4 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2002:72090 CAPLUS
DN 136:118391
TI Novel processes for the preparation of 4-phenylpiperidine derivatives
IN Borrett, Gary Thomas; Fedouloff, Michael; Hughes, Mark Jason; Share, Andrew Colin; Strachan, John Bryce; Szeto, Peter; Voyle, Martyn
PA Smithkline Beecham P.L.C., UK
SO PCT Int. Appl., 34 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2002006275	A1	20020124	WO 2001-GB3221	20010717
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1301508	A1	20030416	EP 2001-949741	20010717
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
JP 2004504319	T2	20040212	JP 2002-512178	20010717

	US 2004087795	A1	20040506	US 2003-333274	20030624
PRAI	GB 2000-17540	A	20000717		
	GB 2000-18857	A	20000801		
	WO 2001-GB3221	W	20010717		

OS CASREACT 136:118391; MARPAT 136:118391

GI



I

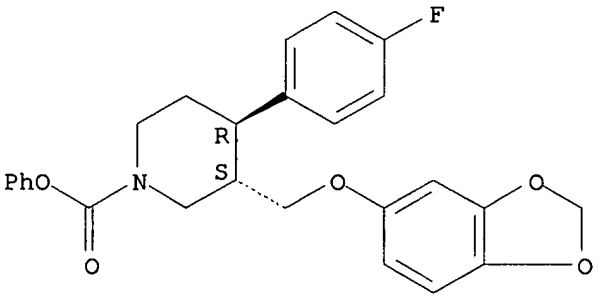
AB A process for preparing a 4-phenylpiperidine I [R = substituted Ph, especially 3,4-methylenedioxyphenyl, R1 = H] from I [R = H, R1 = Me] with or without isolation of intermediate products, comprises reacting I [R = H, R1 = Me] with a sulfonyl chloride, treating the resulting sulfonate with the substituted phenol in the presence of a phase transfer catalyst and a base, treating I [R = substituted Ph, R1 = Me] with a haloformate with addition of an HCl scavenging base, washing the reaction solution containing I [R = substituted Ph, R1 = CO2R2] with an aqueous acid selected from citric acid, phosphoric acid, acetic acid and formic acid, and heating I [R = substituted Ph, R1 = CO2R2] with sodium hydroxide to remove the carbamate group. Preferably the reaction(s) take place in toluene, providing an advantageous procedure for com. production of paroxetine.

IT **253768-88-6P**, (-)-trans-4-(4-Fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)-1-(phenoxycarbonyl)piperidine
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(process for the preparation of 4-phenylpiperidine derivs., such as paroxetine)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 5 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 2001:833279 CAPLUS

DN 135:357848

TI Piperidine compounds and process for providing such

IN Peters, Theodorus Hendricus Antonius; Lemmens, Jacobus Maria; Slanina, Pavel

PA Synthon B.V., Neth.
SO PCT Int. Appl., 23 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001085689	A1	20011115	WO 2000-NL321	20000512
	W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	AU 2000046281	A5	20011120	AU 2000-46281	20000512
	EP 1286965	A1	20030305	EP 2000-927979	20000512
	EP 1286965	B1	20040114		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL				
	AT 257823	E	20040115	AT 2000-927979	20000512
	PT 1286965	T	20040430	PT 2000-927979	20000512
	ES 2209876	T3	20040701	ES 2000-927979	20000512
	NL 1016013	C1	20010130	NL 2000-1016013	20000824
	US 2002099219	A1	20020725	US 2001-853860	20010514
PRAI	EP 2000-927979	A	20000512		
	WO 2000-NL321	A	20000512		

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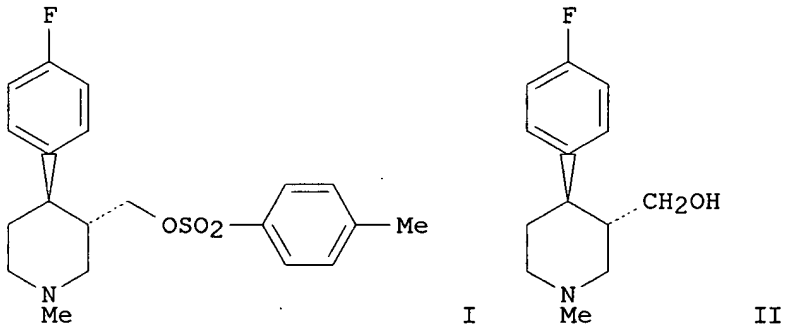


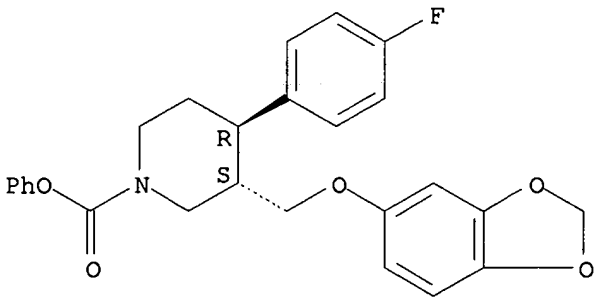
AB The present invention relates to a process for providing a compound of formula I, a hydrate, solvate, and/or salt thereof. Thus, the reaction of II with tosyl chloride in the presence of triethylamine provided tosylate I in 83% crude yield. Compound I was subsequently purified by recrystn. from isopropanol to provide an overall yield of 75%.

IT **253768-88-6P 317323-78-7P**
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(piperidine compds. and process for providing such)

RN 253768-88-6 CAPLUS

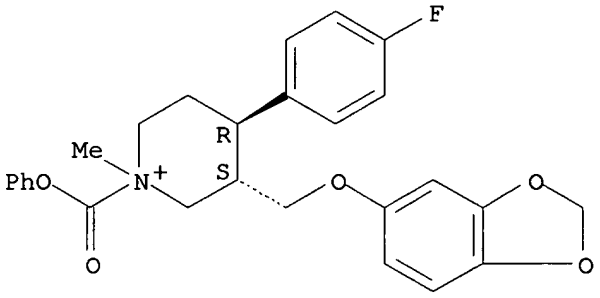
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RN 317323-78-7 CAPLUS
CN Piperidinium, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-1-methyl-1-(phenoxycarbonyl)-, chloride, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



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RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

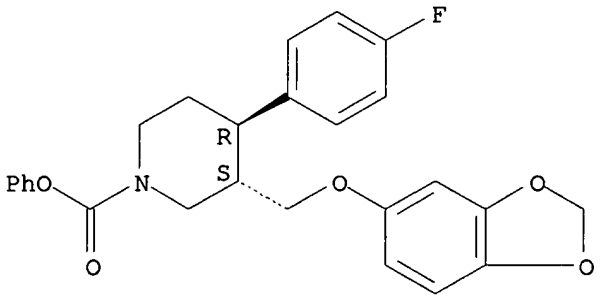
L11 ANSWER 6 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:526388 CAPLUS
DN 135:112014
TI Preparation of noncrystalline paroxetine hydrochloride
IN Craig, Andrew Simon; Jacewicz, Victor Witold
PA Smithkline Beecham Plc, UK
SO U.S. Pat. Appl. Publ., 3 pp., Cont. of U. S. Ser. No. 179,714.
CODEN: USXXCO
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2001008940	A1	20010719	US 2001-759513	20010112
PRAI	GB 1997-22694	A	19971027		
	US 1998-179714	A1	19981027		

AB Non-crystalline paroxetine-HCl is prepared by precipitation as a solid from a solution of paroxetine-HCl, or by drying an oil containing paroxetine-HCl, or by removing water/solvent from a hydrate/solvate. The oil may also be obtained by precipitation from a solution of paroxetine-HCl.

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of noncryst. paroxetine hydrochloride)
RN 253768-88-6 CAPLUS
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

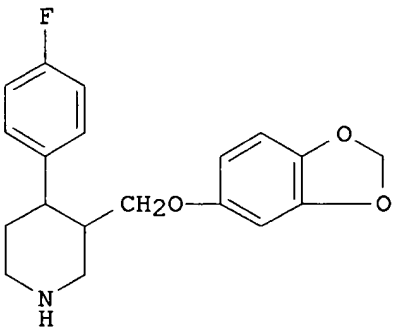
Absolute stereochemistry. Rotation (-).



L11 ANSWER 7 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:300712 CAPLUS
DN 134:311117
TI Novel processes for synthesis of paroxetine
IN Crowe, David; Ward, Neal; Wells, Andrew Stephen
PA Smithkline Beecham Plc, UK
SO PCT Int. Appl., 55 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001029032	A1	20010426	WO 2000-GB4066	20001020
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
PRAI	GB 1999-24882	A	19991020		

OS CASREACT 134:311117; MARPAT 134:311117
GI



I

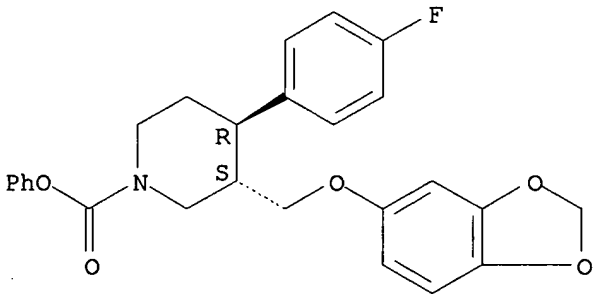
AB Three process schemes for a complete route to paroxetine (I) starting from arecoline are disclosed.

IT **253768-88-6P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(synthesis of paroxetine)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 8 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 2001:300711 CAPLUS

DN 134:311116

TI Process for the preparation of paroxetine

IN Borrett, Gary Thomas; Crowe, David; Ward, Neal; Wells, Andrew Stephen

PA Smithkline Beecham P.L.C., UK

SO PCT Int. Appl., 47 pp.
CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2001029031	A1	20010426	WO 2000-GB4060	20001020

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

PRAI GB 1999-24855 A 19991020

OS MARPAT 134:311116

AB Three process schemes for a complete route to paroxetine from a pyridine ester are disclosed. E.g., enzymic resolution of trans-1-methyl-3-carbomethoxy-4-(4'-fluorophenyl)piperidine is described.

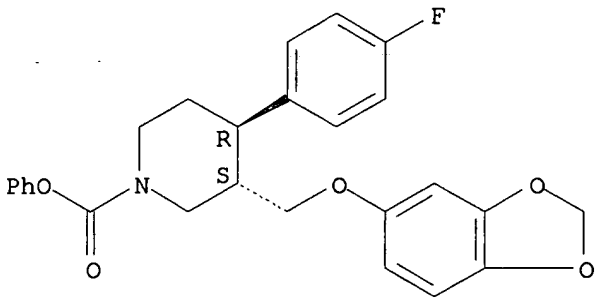
IT **253768-88-6P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of paroxetine)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-

fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 9 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:265415 CAPLUS
DN 134:285600
TI Preparation of paroxetine hydrochloride acetone solvate
IN Craig, Andrew Simon
PA Smithkline Beecham PLC, UK
SO PCT Int. Appl., 13 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2001025232	A1	20010412	WO 2000-GB3802	20001004

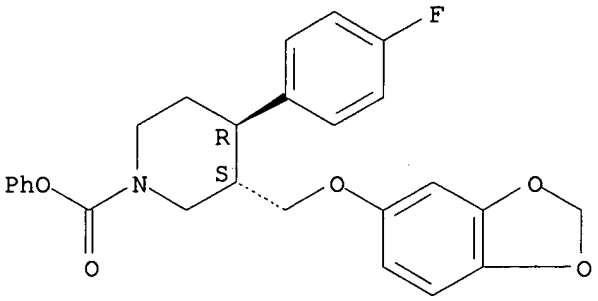
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

PRAI GB 1999-23439 A 19991004

AB A solution of paroxetine base, or a salt of paroxetine with an organic acid, in an organic solvent is treated with aqueous HCl, the solution is then distilled to reduce the amount of water present and then treated with acetone to give paroxetine hydrochloride acetone solvate (I) as a crystalline solid. Concentrate HCl was added to a solution of paroxetine free base in toluene and the mixture heated to 90° for 5 min. One-half of the total volume of the solvent was removed and dry acetone was added to give I.

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of paroxetine hydrochloride acetone solvate)
RN 253768-88-6 CAPLUS
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 10 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:265413 CAPLUS
DN 134:285598
TI Process for the preparation of paroxetine hydrochloride acetone solvate
IN Craig, Andrew Simon
PA Smithkline Beecham PLC, UK
SO PCT Int. Appl., 10 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2001025230	A1	20010412	WO 2000-GB3795	20001004

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

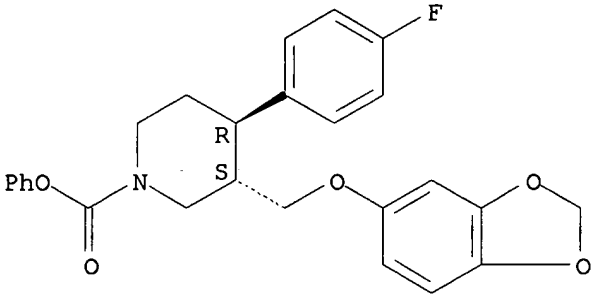
PRAI GB 1999-23445 A 19991004

AB A solution of paroxetine base, or a solution of salt of paroxetine with an organic acid is treated with acetone and a solution of HCl in a carrier solvent, to give paroxetine-HCl acetone solvate (I) as a crystalline solid. Dry acetone was added to the free paroxetine base and HCl in MeOH was added. The product was filtered and treated with acetone and dried at 60° for 20 h. I was characterized by IR spectroscopy.

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of paroxetine hydrochloride acetone solvate)

RN 253768-88-6 CAPLUS
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



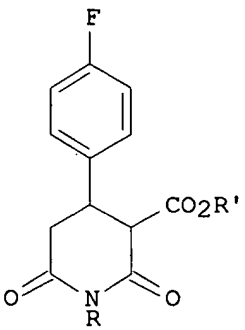
RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 11 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:152646 CAPLUS
DN 134:207718
TI Process for preparation of paroxetine intermediate
IN Crowe, David; Jones, David Alan
PA Smithkline Beecham P.L.C., UK
SO PCT Int. Appl., 20 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2001014335	A1	20010301	WO 2000-EP8177	20000818

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

PRAI GB 1999-20147 A 19990825
OS CASREACT 134:207718; MARPAT 134:207718
GI



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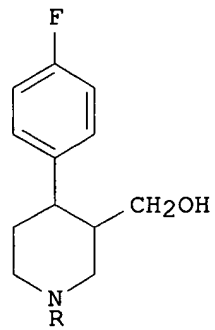


II

AB Trans-piperidinediones I [R = benzyl group; R' is an optionally substituted C1-6-alkyl, aryl-C1-6-alkyl, C1-6-allyl, aryl] were prepared by reaction of a cinnamate ester with a malonamide in the presence of a strong base. Compound II, prepared from I, is used in the presentation of

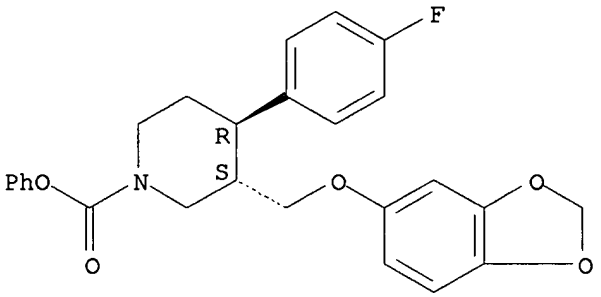
paroxetine. The preparation of paroxetine by this route avoids the formation of difficult to remove impurities found in other routes. Thus, reaction of Me malonyl chloride with PhCH2NH2, followed by reaction with 4-C6H4CHO gave trans-4-(4-fluorophenyl)-1-benzyl-2,6-dioxo-piperidine-3-carboxylic acid Et ester. Reduction of the latter with LiAlH4 and resolution led to (-)-trans-1-benzyl-4-(4-fluorophenyl)-3-hydroxymethylpiperidine. Reaction of the product with MeSO2Cl, then with sesamol followed by Ph chloroformate gave (-)-trans-4-(4-fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)-N-phenoxycarbonylpiperidine. Hydrogenation of the latter in presence of Pd on C gave paroxetine hydrochloride hemihydrate.

IT **253768-88-6P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(process for preparation of paroxetine intermediate)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 12 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 2001:137210 CAPLUS

DN 134:198046

TI Preparation of paroxetine free base

IN Craig, Andrew Simon; Jones, David Alan; O'Keeffe, Deirdre; Ward, Neal

PA SmithKline Beecham P.L.C., UK

SO PCT Int. Appl., 37 pp.
CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001012624	A1	20010222	WO 2000-GB3107	20000811
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	AU 724845	B3	20000928	AU 1999-48821	19990920
PRAI	GB 1999-19052	A	19990812		

AB Processes are disclosed for preparing paroxetine free base in substantially

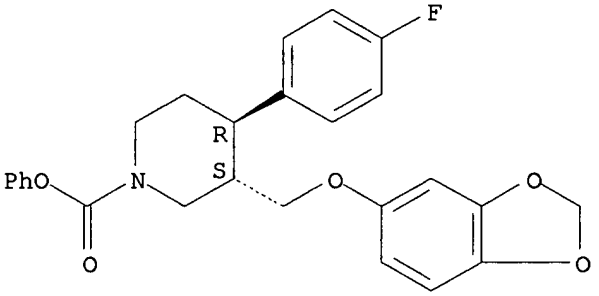
pure form. The free base may be combined with a pharmaceutically acceptable diluent and/or converted in-situ to a pharmaceutically acceptable salt. N-phenoxycarbonyl paroxetine was refluxed with potassium hydroxide in toluene to obtain paroxetine base which was separated and purified.

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of paroxetine free base)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 13 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 2001:50640 CAPLUS

DN 134:115856

TI Preparation of N-substituted 4-(4-fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)piperidines by reaction of the corresponding 3-sulfonyloxymethyl compounds with sesamol or derivatives.

IN Gordon, Alison Ruth

PA SmithKline Beecham PLC, UK

SO PCT Int. Appl., 33 pp.
CODEN: PIXXD2

DT Patent

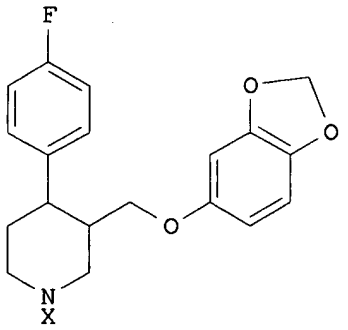
LA English

FAN.CNT 1

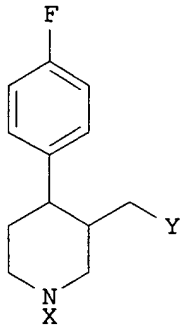
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001004113	A2	20010118	WO 2000-GB2638	20000707
	WO 2001004113	A3	20010712		
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	EP 1246821	A2	20021009	EP 2000-946071	20000707
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL				
	JP 2003504365	T2	20030204	JP 2001-509723	20000707
PRAI	GB 1999-16187	A	19990709		
	WO 2000-GB2638	W	20000707		

OS CASREACT 134:115856; MARPAT 134:115856

GI



I



II

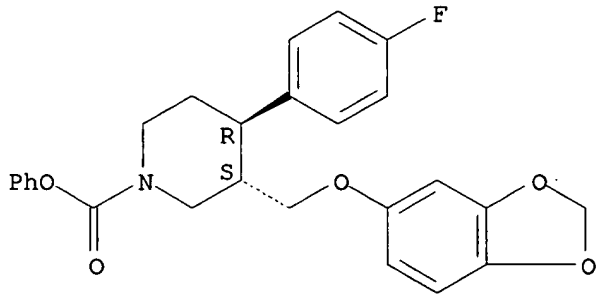
AB Title compds. [I; X = (substituted) alkyl, aralkyl, allyl, alkynyl], were prepared by preparing and reacting [II; X as above; Y = (substituted) alkyl-, aryl-, or aralkylsulfonate] (unisolated) with sesamol or a derivative thereof. Thus, (-)-trans-4-(4-fluorophenyl)-3-hydroxymethyl-1-methylpiperidine and Me2NEt in PhMe at -2° to 2° were treated with PhSO2Cl in PhMe over 70 min. followed by stirring for 20 min. to 10° to give a solution of sulfonate ester in PhMe which was worked up and then combined with DMF, heated to 50°, and treated with sesamol and NaOMe in DMF over 20 min. to give 87.6% (-)-trans-4-(4-fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)-1-methylpiperidine.

IT **253768-88-6P**, (-)-trans-4-(4-Fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)-1-phenoxycarbonylpiperidine
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(preparation of N-substituted 4-(4-fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)piperidines by reaction of the corresponding 3-sulfonyloxymethyl compds. with sesamol or derivs.)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



L11 ANSWER 14 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 2001:28544 CAPLUS

DN 134:86162

TI Piperidine derivative intermediate for paroxetin preparation

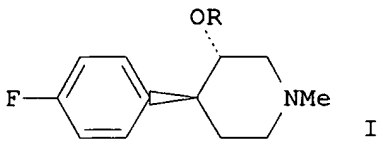
PA Synthon B.V., Neth.

SO Ger. Gebrauchsmusterschrift, 17 pp.

CODEN: GGXXFR
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 20015732	U1	20010111	DE 2000-20015732	20000912
PRAI	DE 2000-20015732		20000912		

OS MARPAT 134:86162
GI



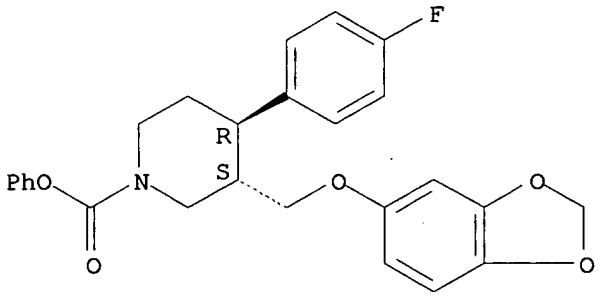
I

AB Piperidinylmethyl tosylate derivative (-)-I (R = Ts) (7.3 g) was prepared by reaction of 5.2 g paroxol [(-)-I, R = H] with 4.7 g tosyl chloride in Et3N-EtOAc.

IT **253768-88-6P 317323-78-7P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

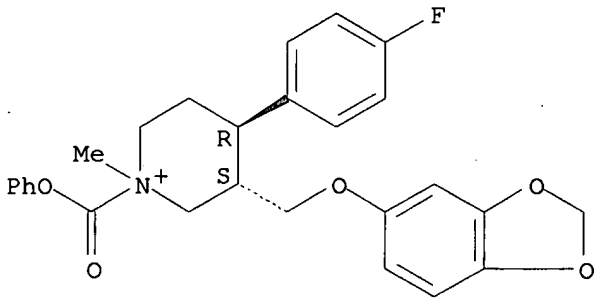
RN 253768-88-6 CAPLUS
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RN 317323-78-7 CAPLUS
CN Piperidinium, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-1-methyl-1-(phenoxycarbonyl)-, chloride, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

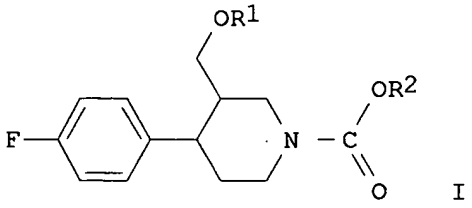


● Cl^-

L11 ANSWER 15 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2000:911248 CAPLUS
DN 134:58215
TI Improved procedure for the manufacture of paroxetine and structurally related compounds
IN Lucas, Edward
PA SmithKline Beecham P.L.C., UK
SO PCT Int. Appl., 24 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2000078753	A1	20001228	WO 2000-GB2455	20000622
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	EP 1187830	A1	20020320	EP 2000-940621	20000622
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	JP 2003502422	T2	20030121	JP 2001-504919	20000622
PRAI	GB 1999-14583	A	19990622		
	WO 2000-GB2455	W	20000622		

OS MARPAT 134:58215
GI



I

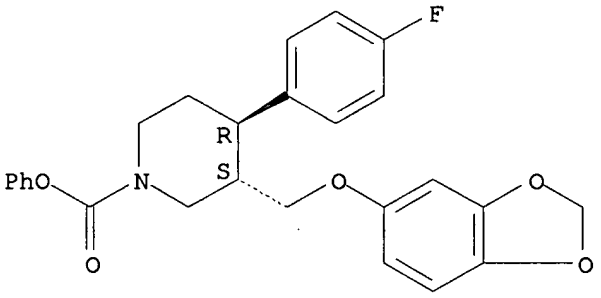
AB 4-(4-Fluorophenyl)piperidine derivs., e.g., the (-)-trans isomer of 4-(4'-fluorophenyl)-3-(3",4"-methylenedioxyphenoxymethyl)piperidine (paroxetine), or their pharmaceutically acceptable salts, useful for the treatment of, e.g., depression, obsessive compulsive disorder and panic, are manufactured by hydrolyzing solns. of carbamate precursors [I; R1 = substituted Ph; R2 = C1-6 alkyl, C3-6 cycloalkyl, aralkyl group, (un)substituted Ph] by heating with a base, e.g., KOH, in a solvent, preferably PhMe, then discontinuing the heating while stirring vigorously to form a finely divided (sand-like) complex derived from the base and the carbamate. The process is carried out under anhydrous or dehydrating conditions, including removal of H2O by azeotropic distillation In previous procedures, the hydrolysis reaction was difficult to complete in a reasonable time because KOH melts at PhMe reflux temperature and forms almost insol. complex mass with paroxetine carbamates. The products are crystallized from PhMe in the presence of a cosolvent, preferably EtOH.

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(alkaline hydrolysis; improved procedure for the manufacture of paroxetine)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 16 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2000:421133 CAPLUS
DN 133:63957
TI Derivative of paroxetine for treatment of CNS disorders.
IN Jones, David Alan
PA Smithkline Beecham Plc, UK
SO PCT Int. Appl., 15 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2000035910	A1	20000622	WO 1999-GB4176	19991210

W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF,

CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

	EP 1137646	A1	20011004	EP 1999-961195	19991210
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	JP 2002532494	T2	20021002	JP 2000-588170	19991210
PRAI	GB 1998-27431	A	19981211		
	WO 1999-GB4176	W	19991210		

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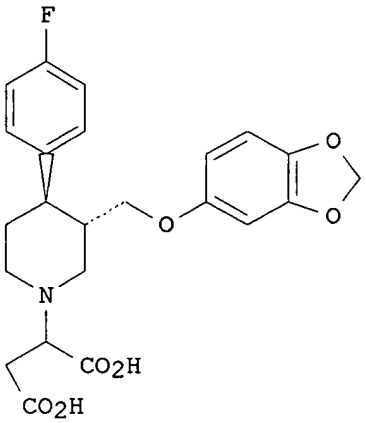


I

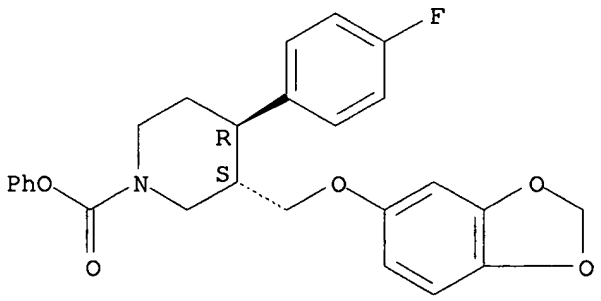
AB I and alkali metal and amine and acid addition salts are useful in the treatment of CNS disorders. Paroxetine was treated with maleic acid to give I paroxetine salt.

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(paroxetine derivative for treatment of CNS disorders)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 17 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 2000:218575 CAPLUS

DN 132:251138

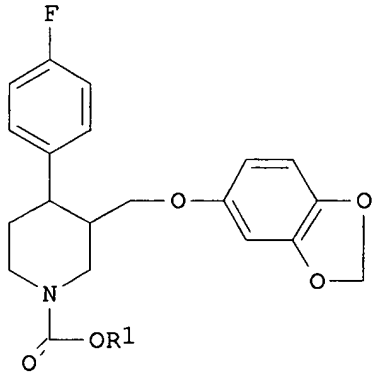
TI Preparation of paroxetine carbamate crystals

IN Nishino, Jiro; Sumiki, Marika; Ohkura, Kazuhiro; Urushibara, Seikou; Wang,

Josho
PA Asahi Glass Co., Ltd., Japan
SO Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DT Patent.
LA Japanese
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 2000095780	A2	20000404	JP 1998-268667	19980922
PRAI	JP 1998-268667		19980922		

OS MARPAT 132:251138
GI

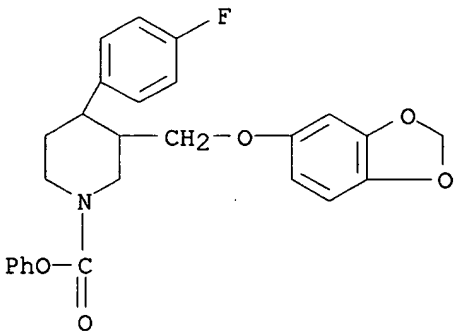


I

AB Crystals of carbamates I [R1 = lower (cyclo)alkyl, lower alkenyl, aralkyl, aryl, heterocyclylalkyl, C1-6 perfluoroalkyl], useful as antidepressants, antiparkinsonian agents, etc. (no data), are prepared by dissolving I into polar solvents and crystallizing I without changing the amts. of the solvents. I (R1 = Et) (20 g, preparation given) was dissolved into aqueous EtOH and cooled at 5° for 30 h to give 18 g white crystals of I (R1 = Et) with 100% purity.

IT **262424-80-6P**
RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)
(preparation of crystals of paroxetine carbamates)

RN 262424-80-6 CAPLUS
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester (9CI) (CA INDEX NAME)



L11 ANSWER 18 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2000:34594 CAPLUS
DN 132:78472
TI Preparation and formulation of paroxetine methanesulfonate
IN Craig, Andrew Simon; Jones, David Alan; O'Keeffe, Deirdre; Ward, Neal
PA SmithKline Beecham PLC, UK
SO Eur. Pat. Appl., 27 pp.
CODEN: EPXXDW
DT Patent
LA English
FAN.CNT 3

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 970955	A1	20000112	EP 1999-303151	19990423
	EP 970955	B1	20000802		
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	CH 689805	A	19991130	CH 1999-723	19990420
	NL 1011874	C1	19990712	NL 1999-1011874	19990423
	GB 2336364	A1	19991020	GB 1999-9505	19990423
	GB 2336364	B2	20000510		
	BE 1011664	A6	19991109	BE 1999-294	19990423
	AU 713131	B3	19991125	AU 1999-23937	19990423
	AU 713877	B3	19991209	AU 1999-23938	19990423
	CA 2269999	AA	20000102	CA 1999-2269999	19990423
	DK 9900554	A	20000103	DK 1999-554	19990423
	FI 9900922	A	20000103	FI 1999-922	19990423
	FI 112077	B1	20031031		
	NO 9901944	A	20000103	NO 1999-1944	19990423
	NO 319030	B1	20050606		
	FR 2780728	A1	20000107	FR 1999-5185	19990423
	FR 2780728	B1	20010216		
	NL 1011875	A1	20000107	NL 1999-1011875	19990423
	NL 1011875	C2	20000324		
	WO 2000001694	A1	20000113	WO 1999-GB1253	19990423
	W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW				
	RW: GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
	AU 9923928	A1	20000120	AU 1999-23928	19990423
	AU 732211	B2	20010412		
	AU 9936191	A1	20000124	AU 1999-36191	19990423
	GB 2339428	A1	20000126	GB 1999-20332	19990423
	DE 19918588	A1	20000127	DE 1999-19918588	19990423
	GR 1003350	B2	20000329	GR 1999-100140	19990423
	GR 99100140	A	20000331		
	ZA 9902899	A	20000329	ZA 1999-2899	19990423
	US 6063927	A	20000516	US 1999-299060	19990423
	PT 102291	A	20000531	PT 1999-102291	19990423
	PT 102291	B	20020731		
	EP 1020463	A1	20000719	EP 2000-201289	19990423
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	AT 195121	E	20000815	AT 1999-303151	19990423
	BE 1012403	A5	20001003	BE 1999-293	19990423
	ES 2149044	T3	20001016	ES 1999-303151	19990423
	PT 970955	T	20001130	PT 1999-303151	19990423

LU 90388 A2 20010129 LU 1999-90388 19990423
GB 2352395 A1 20010131 GB 2000-26487 19990423
GB 2352395 B2 20040211
EP 1089996 A1 20010411 EP 1999-918159 19990423
EP 1089996 B1 20031001
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, FI, RO
TR 200100054 T2 20010621 TR 2001-200100054 19990423
NZ 509180 A 20010629 NZ 1999-509180 19990423
ES 2158778 A1 20010901 ES 1999-849 19990423
ES 2158778 B1 20020316
PT 1020464 T 20011030 PT 2000-201290 19990423
BR 9911682 A 20011226 BR 1999-11682 19990423
GB 2367003 A1 20020327 GB 2001-19695 19990423
IT 1312540 B1 20020422 IT 1999-MI866 19990423
JP 2002519422 T2 20020702 JP 2000-558097 19990423
GB 2377637 A1 20030122 GB 2002-16752 19990423
EP 1288214 A1 20030305 EP 2002-78483 19990423
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, FI, RO, CY
AT 251155 E 20031015 AT 1999-918159 19990423
CN 1127502 B 20031112 CN 1999-810281 19990423
PT 1089996 T 20040227 PT 1999-918159 19990423
ES 2209428 T3 20040616 ES 1999-918159 19990423
IL 140628 A1 20040620 IL 1999-140628 19990423
NL 1012271 A1 19990712 NL 1999-1012271 19990608
NL 1012271 C2 19990923
NL 1012272 C1 19990712 NL 1999-1012272 19990608
CA 2336470 AA 20000113 CA 1999-2336470 19990630
WO 2000001692 A1 20000113 WO 1999-EP4543 19990630
W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW
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AU 9949039 A1 20000124 AU 1999-49039 19990630
EP 1091958 A1 20010418 EP 1999-932772 19990630
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BR 9911679 A 20020129 BR 1999-11679 19990630
JP 2002519421 T2 20020702 JP 2000-558095 19990630
BE 1012420 A6 20001003 BE 1999-832 19991223
EP 1020464 A1 20000719 EP 2000-201290 20000410
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AT 202777 E 20010715 AT 2000-201290 20000410
ES 2157881 T3 20010901 ES 2000-201290 20000410
GR 3034328 T3 20001229 GR 2000-402015 20000905
AU 732558 B3 20010426 AU 2000-69567 20001026
FR 2802098 A1 20010615 FR 2000-14325 20001108
NO 2000006547 A 20010212 NO 2000-6547 20001221
DE 20022645 U1 20020228 DE 2000-20022645 20001228
ZA 2001000020 A 20020325 ZA 2001-20 20010102
BG 105204 A 20011130 BG 2001-105204 20010131
US 2001023252 A1 20010920 US 2001-803798 20010312
US 2001023253 A1 20010920 US 2001-805812 20010314
AU 2001100025 A4 20010621 AU 2001-100025 20010524
GR 3036208 T3 20011031 GR 2001-401061 20010711

	AU 2002100370	A4	20020606	AU 2002-100370	20010711
	US 2002035130	A1	20020321	US 2001-960033	20010921
	HK 1037877	A1	20040723	HK 2001-107165	20011011
	US 2002193406	A1	20021219	US 2002-174380	20020618
	US 2003203937	A1	20031030	US 2003-430026	20030506
	US 2004247667	A1	20041209	US 2004-828660	20040421
PRAI	GB 1998-14316	A	19980702		
	GB 1998-21732	A	19981006		
	GB 1999-2935	A	19990210		
	AU 1999-23928	A3	19990423		
	EP 1999-303151	A3	19990423		
	EP 1999-918159	A3	19990423		
	GB 1999-9505	A3	19990423		
	GB 2000-26487	A3	19990423		
	GB 2001-19695	A	19990423		
	US 1999-299060	A2	19990423		
	WO 1999-GB1253	W	19990423		
	GB 1999-14601	A	19990622		
	GB 1999-14709	A	19990623		
	GB 1999-15096	A	19990628		
	WO 1999-EP4543	W	19990630		
	GB 1999-27501	A	19991119		
	US 1999-454881	B3	19991203		
	US 1999-469902	A1	19991222		
	US 2000-635541	A3	20000810		
	US 2001-803798	B1	20010312		
	US 2003-430026	A1	20030506		

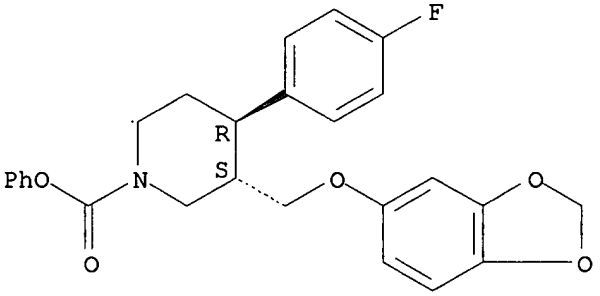
AB The title compound was prepared in several crystallization polymorphs and was used to prepare paroxetine hydrochloride.

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation and formulation of paroxetine methanesulfonate)

RN 253768-88-6 CAPLUS

CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, (3S,4R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (-).



RE.CNT 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L11 ANSWER 19 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

AN 1987:18361 CAPLUS

DN 106:18361

TI Piperidine derivatives having gastrointestinal activity

IN Stemp, Jean Anne; Miller, David; Martin, Roger Thomas

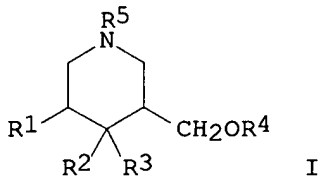
PA Beecham Group PLC, UK

SO Eur. Pat. Appl., 92 pp.
CODEN: EPXXDW

DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 190496	A2	19860813	EP 1985-308936	19851209
	EP 190496	A3	19870527		
	R: BE, CH, DE, FR, GB, IT, LI, LU, NL, SE				
	DK 8505746	A	19860614	DK 1985-5746	19851211
	AU 8551114	A1	19860619	AU 1985-51114	19851211
	JP 61180769	A2	19860813	JP 1985-278125	19851212
PRAI	GB 1984-31478	A	19841213		
	GB 1985-20619	A	19850816		

OS MARPAT 106:18361
GI



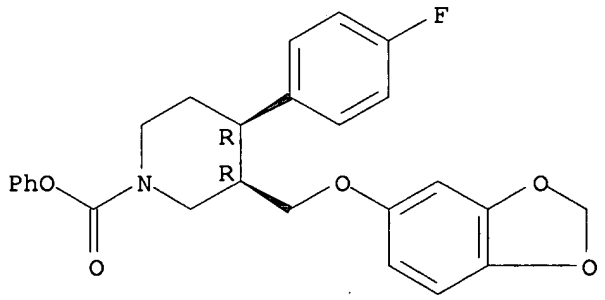
I

AB Title compds. I [R1, R2 = H, R1R2 = bond; R3, R4 = (un)substituted Ph, naphthyl; R5 = (CH2)nR6; R6 = (un)substituted Ph or naphthyl; n = 1, 2] and their salts, useful as antiulcer agents and for treatment of impaired gastrointestinal motility, were prepared Thus, (-)-trans-4-(4-fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)piperidine in DMF was reacted with PhCH2Cl to give (-)-trans-1-benzyl-4-(4-fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)piperidine (II). In antiulcer tests on rats, II at 10.5 mg/kg orally showed 53% inhibition of gastric erosions.

IT **105812-85-9P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of, as intermediate for fluorophenylpiperidine derivative)

RN 105812-85-9 CAPLUS
CN 1-Piperidinecarboxylic acid, 3-[(1,3-benzodioxol-5-yloxy)methyl]-4-(4-fluorophenyl)-, phenyl ester, cis- (9CI) (CA INDEX NAME)

Relative stereochemistry.



=> d his

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FILE 'CAPLUS' ENTERED AT 10:59:50 ON 07 MAR 2006

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L7               STRUC
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L11           19 S L10 NOT L1

=> s l11 and toluen?
        217023 TOLUEN?
L12            4 L11 AND TOLUEN?

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L12  ANSWER 1 OF 4  CAPLUS  COPYRIGHT 2006 ACS on STN
AN   2002:72090  CAPLUS
DN   136:118391
TI   Novel processes for the preparation of 4-phenylpiperidine derivatives
IN   Borrett, Gary Thomas; Fedouloff, Michael; Hughes, Mark Jason; Share,
     Andrew Colin; Strachan, John Bryce; Szeto, Peter; Voyle, Martyn
PA   Smithkline Beecham P.L.C., UK
SO   PCT Int. Appl., 34 pp.
     CODEN: PIXXD2
DT   Patent
LA   English
FAN.CNT 1
     PATENT NO.           KIND   DATE        APPLICATION NO.          DATE
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PI   WO 2002006275         A1    20020124    WO 2001-GB3221           20010717
         W:  AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
             CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM,
             HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS,
             LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO,
             RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ,
             VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY,
             DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
             BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
     EP 1301508             A1    20030416    EP 2001-949741           20010717
         R:  AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, SI, LT, LV, FI, RO, MK, CY, AL, TR
     JP 2004504319          T2    20040212    JP 2002-512178           20010717
     US 2004087795          A1    20040506    US 2003-333274           20030624
PRAI GB 2000-17540          A     20000717
     GB 2000-18857          A     20000801
     WO 2001-GB3221         W     20010717
OS   CASREACT 136:118391; MARPAT 136:118391
RE.CNT  6     THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
              ALL CITATIONS AVAILABLE IN THE RE FORMAT
AB   A process for preparing a 4-phenylpiperidine I [R = substituted Ph, especially
     3,4-methylenedioxyphenyl, R1 = H] from I [R = H, R1 = Me] with or without
     isolation of intermediate products, comprises reacting I [R = H, R1 = Me]
     with a sulfonyl chloride, treating the resulting sulfonate with the
     substituted phenol in the presence of a phase transfer catalyst and a
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base, treating I [R = substituted Ph, R1 = Me] with a haloformate with addition of an HCl scavenging base, washing the reaction solution containing I [R = substituted Ph, R1 = CO2R2] with an aqueous acid selected from citric acid, phosphoric acid, acetic acid and formic acid, and heating I [R = substituted Ph, R1 = CO2R2] with sodium hydroxide to remove the carbamate group. Preferably the reaction(s) take place in **toluene**, providing an advantageous procedure for com. production of paroxetine.

IT 110429-36-2P, (-)-trans-4-(4-Fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)-1-methylpiperidine **253768-88-6P**, (-)-trans-4-(4-Fluorophenyl)-3-(3,4-methylenedioxyphenoxymethyl)-1-(phenoxycarbonyl)piperidine
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(process for the preparation of 4-phenylpiperidine derivs., such as paroxetine)

IT 64-18-6, Formic acid, uses 64-19-7, Acetic acid, uses 77-92-9, Citric acid, uses 108-88-3, **Toluene**, uses 7087-68-5 7664-38-2, Phosphoric acid, uses
RL: NUU (Other use, unclassified); USES (Uses)
(process for the preparation of 4-phenylpiperidine derivs., such as paroxetine)

L12 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:265415 CAPLUS
DN 134:285600
TI Preparation of paroxetine hydrochloride acetone solvate
IN Craig, Andrew Simon
PA Smithkline Beecham PLC, UK
SO PCT Int. Appl., 13 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2001025232	A1	20010412	WO 2000-GB3802	20001004

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

PRAI GB 1999-23439 A 19991004
RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

AB A solution of paroxetine base, or a salt of paroxetine with an organic acid, in an organic solvent is treated with aqueous HCl, the solution is then distilled to reduce the amount of water present and then treated with acetone to give paroxetine hydrochloride acetone solvate (I) as a crystalline solid. Concentrate HCl was added to a solution of paroxetine free base in **toluene** and the mixture heated to 90° for 5 min. One-half of the total volume of the solvent was removed and dry acetone was added to give I.

IT 64-17-5, Ethanol, uses 67-66-3, uses 71-23-8, 1-Propanol, uses 75-05-8, Acetonitrile, uses 75-09-2, Methylene chloride, uses 79-20-9 108-88-3, **Toluene**, uses 109-99-9, THF, uses 123-91-1, Dioxane, uses 141-78-6, EtOAc, uses 142-82-5, Heptane, uses

1330-20-7, Xylene, uses
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
(preparation of paroxetine hydrochloride acetone solvate)
IT 67-64-1, Acetone, reactions **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of paroxetine hydrochloride acetone solvate)

L12 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:137210 CAPLUS
DN 134:198046
TI Preparation of paroxetine free base
IN Craig, Andrew Simon; Jones, David Alan; O'Keeffe, Deirdre; Ward, Neal
PA SmithKline Beecham P.L.C., UK
SO PCT Int. Appl., 37 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2001012624	A1	20010222	WO 2000-GB3107	20000811
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
AU 724845	B3	20000928	AU 1999-48821	19990920
PRAI GB 1999-19052	A	19990812		

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT
AB Processes are disclosed for preparing paroxetine free base in substantially pure form. The free base may be combined with a pharmaceutically acceptable diluent and/or converted in-situ to a pharmaceutically acceptable salt. N-phenoxycarbonyl paroxetine was refluxed with potassium hydroxide in **toluene** to obtain paroxetine base which was separated and purified.
IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of paroxetine free base)

L12 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2000:911248 CAPLUS
DN 134:58215
TI Improved procedure for the manufacture of paroxetine and structurally related compounds
IN Lucas, Edward
PA SmithKline Beecham P.L.C., UK
SO PCT Int. Appl., 24 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2000078753	A1	20001228	WO 2000-GB2455	20000622
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR,				

HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

EP 1187830 A1 20020320 EP 2000-940621 20000622
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO

JP 2003502422 T2 20030121 JP 2001-504919 20000622

PRAI GB 1999-14583 A 19990622

WO 2000-GB2455 W 20000622

OS MARPAT 134:58215

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

IT **253768-88-6**
RL: RCT (Reactant); RACT (Reactant or reagent)
(alkaline hydrolysis; improved procedure for the manufacture of paroxetine)

IT 108-88-3, **Toluene**, uses
RL: TEM (Technical or engineered material use); USES (Uses)
(solvent; improved procedure for the manufacture of paroxetine involving alkaline hydrolysis of (fluorophenyl)piperidine carbamate precursor with hydroxide in)

IT 64-17-5, Ethanol, uses
RL: TEM (Technical or engineered material use); USES (Uses)
(tech., cosolvent; improved procedure for the manufacture of paroxetine involving crystallization from **toluene** containing)